



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C11D	A2	(11) International Publication Number: WO 99/51714 (43) International Publication Date: 14 October 1999 (14.10.99)
(21) International Application Number: PCT/EP99/00493 (22) International Filing Date: 26 January 1999 (26.01.99) (30) Priority Data: 9807477.6 7 April 1998 (07.04.98) GB (71) Applicant (for AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4 4BQ (GB). (71) Applicant (for all designated States except AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG US ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (71) Applicant (for IN only): HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020, Maharashtra (IN). (72) Inventors; and (75) Inventors/Applicants (for US only): DARU, Vijay [IN/IN]; Hindustan Lever Ltd., Research Centre, Chakala, Andheri (East), Mumbai 400 099 (IN). PEREIRA, Winston, Anthony [IN/IN]; Hindustan Lever Ltd., Research Centre, Chakala,		Andheri (East), Mumbai 400 099 (IN). SENGUPTA, Rana [IN/IN]; Hindustan Lever Ltd., Survey No. 907, Kilwani Road, Amli Village, Silvassa, PIN - 396230, Union Territory of Dadra & Nagar Haveli (IN). (74) Agent: FRANSELLA, Mary, Evelyn; Unilever plc, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>Without international search report and to be republished upon receipt of that report.</i>
(54) Title: COLOURED GRANULAR COMPOSITION FOR USE IN PARTICULATE DETERGENT COMPOSITIONS (57) Abstract <p>A granular composition suitable for incorporation into a particulate detergent composition comprises: (a) a coloured or fluorescent ingredient, for example, a photobleach such as zinc or aluminium phthalocyanine sulphonate; (b) a carrier material for the coloured or fluorescent ingredient which is an α-hydroxy organic acid, preferably citric acid; (c) a water-soluble or water-dispersible barrier material, preferably a water-soluble starch or polyvinyl alcohol; and (d) an inorganic flow modifier, for example, silica and/or china clay. Preferably, the coloured or fluorescent ingredient is dispersed onto the α-hydroxyacid which is mixed with part or all of the inorganic flow modifier and the whole treated with the barrier material, then granulated with any remaining inorganic flow modifier. The granular material can be incorporated into particulate detergent compositions, where it shows a reduced tendency to leave stains on washed fabrics.</p>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

COLOURED GRANULAR COMPOSITION FOR USE IN
PARTICULATE DETERGENT COMPOSITIONS

5

Technical field

The present invention relates to a fast-dissolving, low-staining granular composition containing a visually
10 distinct, eg coloured or fluorescent, ingredient, for example, a photobleach. The invention more particularly relates to granules containing a coloured or fluorescent ingredient which may be incorporated into a detergent composition without encountering the problem of staining of
15 the fabric during the wash.

Background and Prior Art

20 Coloured speckles are used in detergent formulations to give a distinct appearance to the product as well as to deliver functional benefits. In the wash process, it is undesirable that these coloured speckles settle on to the fabric surface leading to staining of the fabric. It has also been noticed
25 that these stains do not get washed out in subsequent laundering. Further the uneven concentration of these speckles on the substrate could result in localised higher levels of the active causing fabric damage.

30 US 4 097 418 (Procter & Gamble, 1978) discloses a granular coloured speckle having non-staining qualities comprising an agglomerate of a granular water soluble inorganic alkaline salt with an anionic surfactant which contains a dye or

- 2 -

pigment. Mixing of the dye and the anionic surfactant prior to agglomeration prevents the pigment particles from attaching to each other and on to the fabric surface thus preventing staining of the fabric.

5

US 4 762 636 (Ciba-Geigy, 1988) discloses a process for obtaining granules with defined physical properties that float on an aqueous treatment liquor and release the active ingredient without getting deposited on to the substrate.

10

Summary of the invention

15 It is thus an object of the present invention to provide a granule composition containing a coloured or fluorescent ingredient which may be incorporated into a detergent composition without encountering the problem of staining of the fabric during the wash process.

20 It is a further object of the invention to provide a granule composition having a superior non-staining property, which may be manufactured by a process which is more economical and would involve lower capital cost as compared to the known processes.

25

Accordingly the present invention provides a granular composition suitable for incorporation into a particulate detergent composition, the granular composition comprising

30 (a) a coloured or fluorescent ingredient,

- 3 -

(b) an α -hydroxy organic acid as a carrier material for the coloured or fluorescent ingredient,

(c) a water-soluble or water-dispersible barrier material,
5 and

(d) an inorganic flow modifier.

The water-soluble or water-dispersible barrier material
10 serves to provide a protective layer or coating on the combination of the coloured or fluorescent ingredient and its carrier material, the α -hydroxy organic acid.

Preferably at least a part of the inorganic flow modifier is also thus protected. Additional inorganic flow modifier
15 that is not protected by the barrier material may also be present.

The term coating used above does not necessarily imply complete coating.

20

According to a preferred embodiment of the invention, therefore,

- the coloured or fluorescent ingredient is dispersed
25 onto, or in the form of a coating over, the α -hydroxy organic acid,

- the α -hydroxy organic acid carrying the coloured or fluorescent ingredient is in admixture with the flow
30 modifier or a portion thereof,

- 4 -

- the barrier material is in the form of a coating over the said mixture of α -hydroxy organic acid and flow modifier, being combined with (in admixture with) any remaining portion of the flow modifier.

5

According to a further preferred aspect of the invention the granule composition comprises:

- from 40 to 90% by weight, preferably from 60 to 80% by weight, of the α -hydroxy organic acid,

10

- from 0.5 to 5% by weight, preferably from 1 to 2% by weight, of the coloured or fluorescent ingredient,

15

- from 10 to 50% by weight, preferably from 18 to 40% by weight, of the inorganic flow modifier, and

- from 0.5 to 5% by weight, preferably from 1 to 3% by weight, of the water soluble barrier material.

20

The granular composition of the invention may be prepared by a process in which the coloured or fluorescent ingredient is dispersed onto the α -hydroxyacid, which is then mixed with part or all of the inorganic flow modifier and the whole treated with the barrier material, then granulated with any remaining inorganic flow modifier.

25

According to a further aspect of the invention, therefore, there is provided a process for the preparation of a granule composition which comprises the steps of:

30

- 5 -

- (i) dispersing the coloured or fluorescent ingredient on the α -hydroxy acid and granulating with the inorganic flow modifier or a portion thereof;
- 5 (ii) drying the material, preferably at a temperature within the range of from 15 to 50°C, more preferably from 20 to 40°C;
- (iii) treating the dried material with the water-soluble or
10 water-dispersible barrier material;
- (iv) optionally, granulating the said treated material with any remaining flow aid modifier and drying.
- 15 The α -hydroxy organic acid may be selected from citric acid, adipic acid, tartaric acid, maleic acid and others. An especially preferred acid is citric acid.

Examples of coloured or fluorescent ingredients are
20 photoactivators (also known as photobleaches), especially phthalocyanines; coloured dyes and pigments; fluorescent whitening agents; and mixtures thereof. Especially preferred ingredients are the photobleaches, zinc and aluminium phthalocyanine sulphonates.

25 The inorganic flow modifier can be selected from china clay, (preferably washed china clay), silica, precipitated silica, precipitated calcium carbonate and diatomaceous/siliceous earth. China clay, silica and mixtures thereof are
30 especially preferred.

- 6 -

The water-soluble or water-dispersible, preferably water-soluble, barrier material may suitably be selected from water-soluble starches, polyvinyl alcohols, polyethylene glycols and latex. Preferred materials are starches and
5 polyvinyl alcohols.

As previously indicated, the granules of the present invention may be prepared by dispersing the coloured or fluorescent ingredient on the α -hydroxy organic acid, for
10 example, in a rotary pan granulator, and subsequently or simultaneously adding a suitable flow modifier. The resultant agglomerate is dried and further coated with the water-soluble or water-dispersible barrier material, and optionally granulated with further flow modifier and dried.

15 A further subject of the invention is a particulate detergent composition comprising organic detergent surfactants and other detergent ingredients, and comprising a granular composition as defined previously. The
20 particulate laundry detergent composition of the invention may suitably comprise from 5 to 60 wt% of organic surfactant, from 10 to 80 wt% of detergency builder, from 0.1 to 10 wt%, preferably from 0.5 to 5 wt%, of a granular composition as defined previously, and optionally other
25 detergent ingredients to 100 wt%.

The invention will now be illustrated further by means of the following non-limiting Examples, in which parts and percentages are by weight unless otherwise stated. Examples
30 denoted by a number are in accordance with the invention, while Examples denoted by a letter are comparative.

- 7 -

EXAMPLE 1, COMPARATIVE EXAMPLES A TO CPreparation of granular compositions5 Example 1: photobleach granules using citric acid carrier
and starch barrier material

8 Kg of citric acid were placed in a rotary pan granulator and 200 g of aluminium phthalocyanine sulphonate solution
10 (10% concentration) were sprayed on to it. During spraying, half the quantity of a mixture of 0.8 kg of china clay and 500 g of silica was added. The material was dried at 40°C for about 4 hours. The material was taken back into a rotary pan granulator and 500 g of starch solution
15 (concentration 10% by weight) was sprayed on. The balance half of the mixture of china clay and silica was added. The material was dried at 40°C for 4 hours.

The formulation of the granular material was as follows:

20

	Starting material (g)	Final formulation (wt%)
Aluminium phthalocyanine sulphonate	20	2
Citric acid	8000	80
China clay	800	8
Silica	500	5
Starch	50	0.5
Water to 100%	630	4.5

- 8 -

Example 2: photobleach granules using citric acid carrier
and polyvinyl alcohol barrier material

Photobleach granules were prepared as in Example 1 but the
5 starch solution was replaced by polyvinyl alcohol solution
(concentration 10% by weight).

The final formulation of the granular material was as
follows:

10

	Starting material (g)	Final formulation (wt%)
Aluminium phthalocyanine sulphonate	20	2
Citric acid	8000	80
China clay	800	8
Silica	500	5
Polyvinyl alcohol	50	0.5
Water to 100%	630	4.5

Comparative Example A

15

Granules were prepared by the process of Example 1 but using
soda ash instead of citric acid.

20 Comparative Example B

Granules were prepared by the process of Example 1 but using
sodium tripolyphosphate instead of citric acid.

- 9 -

Wash protocol

Cotton fabric pieces of dimension 10 inch x 10 inch (25.4 cm x 25.4 cm) were placed in trays, each together with 2 kg of water and one of the following photobleach granular compositions:

Example 1

Comparative Example A

10 Comparative Example B

Comparative Example C: commercially available blue speckles, Laundrosil (Trade Mark) PRT-2 ex Sud-Chemie, containing aluminium phthalocyanine sulphonate on a bentonite clay carrier.

15

Each fabric piece was allowed to soak for 30 minutes. Later the fabric was washed in running water and dried in the sun. Four replications were maintained for each treatment. A panel of trained members analysed the fabric visually for blue stains on a scale of 0 to 5 as follows:

Scores	Staining level
25	0-1 minimal/negligible
	2-3 perceptible
	4-5 significant blue staining

30 The data were analysed statistically and are shown in the following Table.

- 10 -

Example	Carrier material in photobleach granule	Average score
1	citric acid	0.9
A	soda ash	2.9
B	sodium tripolyphosphate	4.8
C	commercial	4.5
CD @ 99%		0.71

- 5 The data presented in the table show that the commercially available granules stained the fabric considerably while the use of the granules according to the invention had a significantly superior effect in controlling the staining of the fabric. This is further substantiated by the fact that
- 10 granules made by the same process but by replacing the citric acid with soda ash or sodium tripolyphosphate did not prevent staining of the fabric significantly.

15 EXAMPLES 3 and 4: DETERGENT COMPOSITIONS

Granular detergent compositions were prepared by dry mixing to the following formulations:

20	Linear alkylbenzene sulphonate	30
	Sodium carbonate	20
	Sodium tripolyphosphate	40
	Photobleach granules (see below)	2
	Enzymes, perfume, fluorescer, water etc to	100

25

- 11 -

The detergent composition of Example 3 contained the photobleach granules of Example 1 (citric acid carrier, starch barrier material).

- 5 The detergent composition of Example 4 contained the photobleach granules of Example 2 (citric acid carrier, polyvinyl alcohol barrier material).

- Both detergent compositions were stored in 500 g pouches
10 from May to December under the following conditions representing summer, monsoon and winter conditions in India:

	Temperature (°C)	Relative Humidity (%)
Room temperature	35	80
Hot and dry	42	45
Hot and humid	42	85

15

No instability, as determined by visual inspection, could be detected at the end of the storage period.

20

*

*

*

- 12 -

CLAIMS

- 1 A granular composition suitable for incorporation into
5 a particulate detergent composition, the granular
composition comprising
- (a) a coloured or fluorescent ingredient,
- 10 (b) a carrier material for the coloured or fluorescent
ingredient,
- characterised in that the carrier material (b) is an
 α -hydroxy organic acid, and the granular composition further
15 comprises
- (c) a water-soluble or water-dispersible barrier material,
and
- 20 (d) an inorganic flow modifier.

- 13 -

2 A granular composition as claimed in claim 1,
characterised in that:

- the coloured or fluorescent ingredient (a) is dispersed
5 onto, or in the form of a coating over, the α -hydroxyacid
 (b), which is in admixture with the inorganic flow modifier
 (d) or a portion thereof, and
- the barrier material (c) is in the form of a coating
10 over the mixture of coated α -hydroxyacid and inorganic flow
 modifier, the coated mixture being in admixture with any
 remaining inorganic flow modifier (d).

15 3 A granular composition as claimed in any preceding
 claim, characterised in that it comprises:

- (a) from 0.5 to 5% by weight, preferably from 1 to 2% by
weight, of the coloured or fluorescent ingredient,
20
- (b) from 40 to 90% by weight, preferably from 60 to 80% by
weight, of the α -hydroxy organic acid,
- (c) from 0.5 to 5% by weight, preferably from 1 to 3% by
25 weight, of the water-soluble or water-dispersible barrier
 material.
- (d) from 10 to 50% by weight, preferably from 18 to 40% by
weight, of the inorganic flow modifier.

30

- 14 -

4 A granular composition as claimed in any preceding
claim, characterised in that the coloured or fluorescent
ingredient (a) is selected from photoactivators
(photobleaches), pigments, dyes, and fluorescent whitening
5 agents.

5 A granular composition as claimed in claim 4,
characterised in that the coloured or fluorescent ingredient
10 (a) is a zinc or aluminium phthalocyanine sulphonate.

6 A granular composition as claimed in any preceding
claim, characterised in that the α -hydroxyacid (b) is
15 selected from citric acid, adipic acid, maleic acid and
tartaric acid.

7 A granular composition as claimed in claim 6,
20 characterised in that the α -hydroxyacid (b) is citric acid.

8 A granular composition as claimed in any preceding
claim, characterised in that the water-soluble or water-
25 dispersible barrier material (c) is selected from water-
soluble starches, polyvinyl alcohols, polyethylene glycols,
and latices.

30 9 A granular composition as claimed in claim 8,
characterised in that the barrier material (c) is water-
soluble and is a starch or a polyvinyl alcohol.

- 15 -

10 A granular composition as claimed in any preceding
claim, characterised in that the inorganic flow modifier (c)
is selected from china clay, silica, precipitated silica,
precipitated calcium carbonate, and diatomaceous or
5 siliceous earth.

11 A granular composition as claimed in claim 10,
characterised in that the inorganic flow modifier (c)
10 comprises china clay, silica or a mixture thereof.

12 A particulate laundry detergent composition which
comprises from 5 to 60 wt% of organic surfactant, from 10 to
15 80 wt% of detergency builder, and optionally other detergent
ingredients to 100 wt%, characterised in that it comprises
from 0.1 to 10 wt%, preferably from 0.5 to 5 wt%, of a
granular composition as claimed in any one of claims 1 to
11.

20

- 16 -

13 A process for the preparation of a granular composition as claimed in claim 1, characterised in that it comprises the steps of:

- 5 (i) dispersing the coloured or fluorescent ingredient (a) onto the α -hydroxyacid (b), and granulating with a portion or the whole of the inorganic flow modifier (d),
- 10 (ii) drying the granular material thus obtained,
- (iii) treating the dried material with the water-soluble or water-dispersible barrier material (c),
- 15 (iv) optionally, granulating the treated material thus obtained with any remaining inorganic flow modifier (d) and drying.

20

14 A process as claimed in claim 13, characterised in that the drying step (iii) is carried out at a temperature within the range of from 15 to 50°C, preferably from 20 to 40°C.

25

*

*

*



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁶ : C11D 17/00, 11/00, 3/40, 3/42, 7/26, 3/20	A3	(11) International Publication Number: WO 99/51714 (43) International Publication Date: 14 October 1999 (14.10.99)
(21) International Application Number: PCT/EP99/00493 (22) International Filing Date: 26 January 1999 (26.01.99) (30) Priority Data: 9807477.6 7 April 1998 (07.04.98) GB (71) Applicant (for AU BB CA CY GB GD GH GM IE IL KE LC LK LS MN MW NZ SD SG SL SZ TT UG ZW only): UNILEVER PLC [GB/GB]; Unilever House, Blackfriars, London EC4 4BQ (GB). (71) Applicant (for all designated States except AU BB CA CY GB GD GH GM IE IL IN KE LC LK LS MN MW NZ SD SG SL SZ TT UG US ZW): UNILEVER N.V. [NL/NL]; Weena 455, NL-3013 AL Rotterdam (NL). (71) Applicant (for IN only): HINDUSTAN LEVER LIMITED [IN/IN]; Hindustan Lever House, 165/166 Backbay Reclamation, Mumbai 400 020, Maharashtra (IN). (72) Inventors; and (75) Inventors/Applicants (for US only): DARU, Vijay [IN/IN]; Hindustan Lever Ltd., Research Centre, Chakala, Andheri (East), Mumbai 400 099 (IN). PEREIRA, Winston, Anthony [IN/IN]; Hindustan Lever Ltd., Research Centre, Chakala,		Andheri (East), Mumbai 400 099 (IN). SENGUPTA, Rana [IN/IN]; Hindustan Lever Ltd., Survey No. 907, Kilwani Road, Amli Village, Silvassa, PIN - 396230, Union Territory of Dadra & Nagar Haveli (IN). (74) Agent: FRANELLA, Mary, Evelyn; Unilever plc, Patent Dept., Colworth House, Sharnbrook, Bedford MK44 1LQ (GB). (81) Designated States: AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, US, UZ, VN, YU, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i> <i>Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i> (88) Date of publication of the international search report: 18 November 1999 (18.11.99)
(54) Title: COLOURED GRANULAR COMPOSITION FOR USE IN PARTICULATE DETERGENT COMPOSITIONS		
(57) Abstract A granular composition suitable for incorporation into a particulate detergent composition comprises: (a) a coloured or fluorescent ingredient, for example, a photobleach such as zinc or aluminium phthalocyanine sulphonate; (b) a carrier material for the coloured or fluorescent ingredient which is an α -hydroxy organic acid, preferably citric acid; (c) a water-soluble or water-dispersible barrier material, preferably a water-soluble starch or polyvinyl alcohol; and (d) an inorganic flow modifier, for example, silica and/or china clay.		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AL	Albania	ES	Spain	LS	Lesotho	SI	Slovenia
AM	Armenia	FI	Finland	LT	Lithuania	SK	Slovakia
AT	Austria	FR	France	LU	Luxembourg	SN	Senegal
AU	Australia	GA	Gabon	LV	Latvia	SZ	Swaziland
AZ	Azerbaijan	GB	United Kingdom	MC	Monaco	TD	Chad
BA	Bosnia and Herzegovina	GE	Georgia	MD	Republic of Moldova	TG	Togo
BB	Barbados	GH	Ghana	MG	Madagascar	TJ	Tajikistan
BE	Belgium	GN	Guinea	MK	The former Yugoslav Republic of Macedonia	TM	Turkmenistan
BF	Burkina Faso	GR	Greece	ML	Mali	TR	Turkey
BG	Bulgaria	HU	Hungary	MN	Mongolia	TT	Trinidad and Tobago
BJ	Benin	IE	Ireland	MR	Mauritania	UA	Ukraine
BR	Brazil	IL	Israel	MW	Malawi	UG	Uganda
BY	Belarus	IS	Iceland	MX	Mexico	US	United States of America
CA	Canada	IT	Italy	NE	Niger	UZ	Uzbekistan
CF	Central African Republic	JP	Japan	NL	Netherlands	VN	Viet Nam
CG	Congo	KE	Kenya	NO	Norway	YU	Yugoslavia
CH	Switzerland	KG	Kyrgyzstan	NZ	New Zealand	ZW	Zimbabwe
CI	Côte d'Ivoire	KP	Democratic People's Republic of Korea	PL	Poland		
CM	Cameroon	KR	Republic of Korea	PT	Portugal		
CN	China	KZ	Kazakhstan	RO	Romania		
CU	Cuba	LC	Saint Lucia	RU	Russian Federation		
CZ	Czech Republic	LI	Liechtenstein	SD	Sudan		
DE	Germany	LK	Sri Lanka	SE	Sweden		
DK	Denmark	LR	Liberia	SG	Singapore		
EE	Estonia						

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 99/00493

A. CLASSIFICATION OF SUBJECT MATTER

IPC 6 C11D17/00 C11D11/00 C11D3/40 C11D3/42 C11D7/26
C11D3/20

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C11D B01J

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 3 850 833 A (KOCEICH M ET AL) 26 November 1974 (1974-11-26) claims 1,3-6,11,12 examples 1-3 ---	1,3-5,8, 9,13,14
A	US 4 762 636 A (BALLIELLO PAOLO ET AL) 9 August 1988 (1988-08-09) cited in the application claims 1-6,9,10,12-20 examples ---	1,3-5, 12-14
A	WO 97 33959 A (AMWAY CORP) 18 September 1997 (1997-09-18) examples 1-4 --- -/--	1-4,6,7, 10,11, 13,14

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents :

- "A" document defining the general state of the art which is not considered to be of particular relevance
- "E" earlier document but published on or after the international filing date
- "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- "O" document referring to an oral disclosure, use, exhibition or other means
- "P" document published prior to the international filing date but later than the priority date claimed

- "T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
- "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
- "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.
- "&" document member of the same patent family

Date of the actual completion of the international search

9 September 1999

Date of mailing of the international search report

01 10. 1999

Name and mailing address of the ISA

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

Authorized officer

Neys, P

INTERNATIONAL SEARCH REPORT

International Application No
PCT/EP 99/00493

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P,A	WO 98 16615 A (PROCTER & GAMBLE) 23 April 1998 (1998-04-23) claims examples abstract page 2, line 19 - page 3, line 11 page 12, line 25 - page 13, line 32 -----	1-5, 12-14

INTERNATIONAL SEARCH REPORT

International application No.
PCT/EP 99/00493

Box I Observations where certain claims were found unsearchable (Continuation of item 1 of first sheet)

This International Search Report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.:
because they relate to subject matter not required to be searched by this Authority, namely:
2. ☒ Claims Nos.: 6
because they relate to parts of the International Application that do not comply with the prescribed requirements to such an extent that no meaningful International Search can be carried out, specifically:
see FURTHER INFORMATION sheet PCT/ISA/210
3. ☐ Claims Nos.:
because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box II Observations where unity of invention is lacking (Continuation of item 2 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this International Search Report covers all searchable claims.
2. ☐ As all searchable claims could be searched without effort justifying an additional fee, this Authority did not invite payment of any additional fee.
3. ☐ As only some of the required additional search fees were timely paid by the applicant, this International Search Report covers only those claims for which fees were paid, specifically claims Nos.:
4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this International Search Report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest

- ☐ The additional search fees were accompanied by the applicant's protest.
- ☐ No protest accompanied the payment of additional search fees.

FURTHER INFORMATION CONTINUED FROM PCT/ISA/ 210

Continuation of Box 1.2

Claims Nos.: 6 partially

The application discloses a granule comprising an alpha-hydroxy organic acid as a carrier. In claim 6 a list of acids is given which can function as a carrier. Among these are adipic acid and maleic acid, which are outside the class of alpha-hydroxy organic acids. Therefor, I did not include these acids in my search. However, I searched for malic acid instead of maleic acid, since this could be a typing error.

The applicant's attention is drawn to the fact that claims relating to inventions in respect of which no international search report has been established need not be the subject of an international preliminary examination (Rule 66.1(e) PCT). The applicant is advised that the EPO policy when acting as an International Preliminary Examining Authority is normally not to carry out a preliminary examination on matter which has not been searched. This is the case irrespective of whether or not the claims are amended following receipt of the search report or during any Chapter II procedure.

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/EP 99/00493

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 3850833 A	26-11-1974	AT 319442 B	27-12-1974
		AU 473970 B	08-07-1976
		AU 3822572 A	26-07-1973
		CA 943428 A	12-03-1974
		CH 569083 A	14-11-1975
		DE 2203141 A	17-08-1972
		FR 2123356 A	08-09-1972
		GB 1378923 A	27-12-1974
		NL 7201035 A,B,	27-07-1972
		BE 778472 A	16-05-1972
		IE 36019 B	21-07-1976
		IT 949682 B	11-06-1973
		JP 54119505 A	17-09-1979
		JP 56017400 B	22-04-1981
		PH 12775 A	17-08-1979
		SE 414049 B	07-07-1980
		SE 423406 B	03-05-1982
		SE 7506189 A	30-05-1975
		ZA 7200436 A	26-09-1973
US 4762636 A	09-08-1988	AU 594434 B	08-03-1990
		AU 6952687 A	03-09-1987
		BR 8700957 A	22-12-1987
		EP 0236270 A	09-09-1987
		JP 62207400 A	11-09-1987
		PT 84359 A,B	01-03-1987
WO 9733959 A	18-09-1997	AU 1990697 A	01-10-1997
		CA 2248994 A	18-09-1997
		EP 0888425 A	07-01-1999
WO 9816615 A	23-04-1998	NONE	

